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CITATION:

Kiyama, Ryo ...[et al]. Distribution of the particles in emulsion prepared by high pressure. The Review of Physical Chemistry of Japan 1951, 21: 82-85

ISSUE DATE:

1951

URL:

http://hdl.handle.net/2433/46657

The Review of Physical Chemistry of Japan Vol. 21 No. 1 (1950)

DISTRIBUTION OF THE PARTICLES IN EMULSION PREPARED BY HIGH PRESSURE.

By Ryo Kiyama, Hideo Kinoshita* and Keizo Suzuki.

Introduction.

Sibree[1] prepared the emulsion of paraffin by means of the 2000 lb/in^2 viscoliser, and for the purpose of intravenous infusion of fat, McKibbin and coworkers[2] and Geyer and coworkers[3] prepared the emulsion, which contained harmless emulsifying agents, by means of 4000 lb/in^2 viscoliser. The diameter of the particle is measured by the photomicrographic method, and it was reported by Geyer that it was smaller than $2\sim3\,\mu$. The authors spouted the cod-liver oil in water at higher pressure (1250 atm) and studied the effects of pressure, the diameter of nozzle and crashing plate on the distribution of particle size. In this experiment, tey used the mixture of 30 cc cod-liver oil, 70 cc water and 3 g lecithin, but it is possible to prepare the more concentrated emulsion.

Apparatus and experimental method.

The apparatus used is shown in Fig. 1. Special steel is used for the intensifier B, piston composed C and D, and nozzle G. The area ratio of C and D is 1 : 2.5. A, air compressor, H, circulating pump, J and K, high pressure valves. First of all, push up the piston by pressing the cod-liver oil in F by H. And compressing the air in E by A, and by opening the valve K, the intensified cod-liver oil is spouted in water through the nozzle. There placed the crashing plate made by special steel near the exit of the nozzle. For the purpose of homogenizing the particles of emulsion, emulsion is compressed in F by a circulating pump and the above process is repeated. The experiments under the condition given in

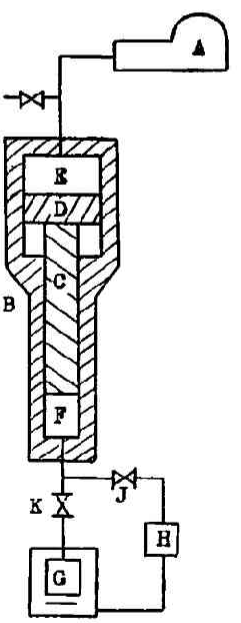

Fig. 1

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1) J. O. Sibree, *Trans. Farad. Soc.*, 27, 161 (1931)
2) J. M. McKibbin, *J. Lab. Clin. Med.*, 30, 488 (1945)
3) R. P. Geyer, *ibid.*, 33, 153 (1948)

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Table 1, where pressure is 250 and 1250 atm. and nozzle diameter is 0.42, 0.50, 1.00 and 1.16 mm, are performed. The direction of the nozzle is perpendicular (direction of FKG) ↓ or horizontal (at right angle to FKG) ↳. The distance between the crashing plate and the exit of nozzle is 2 and 20 mm.

Table 1

Exp. No.	Press. (atm)	Nozzle		Distance of nozzle and crashing plate, (mm)
		Dia. (mm)	Direction	
1	250	1.16	↳	2
2	1250	〃	〃	2
3	〃	0.50	〃	2
4	〃	〃	〃	20
5	〃	1.00	↓	2
6	〃	〃	〃	20
7	〃	0.42	〃	2

Samples.

Cod-liver oil of Japan pharmacopoeia is used, and the lecithin purified from soy bean phosphatid. The purification method of lecithin is as follows: extract with ether and precipitate with aceton, and extract with methanol. After distilating the methanol in evacuation, extract with ether and precipitate with aceton, and repeat this procedure two times.

Method for the determination of the distribution of particle size by centrifugal method.

In order to investigate the preparation of emulsion under various conditions above mentioned, the determination of the distribution of particle size is carried out. The centrifugal method was performed by Svedverg and coworkers[4], but in this experiment the simple and convenient method from the principle of sedimentation analysis[5] is taken up in attention to the facts that the oil particles are separated from medium under the effect of centrifugal force and the clear boundary is formed.

The radius of particle (r) is given as follows from Stokes' law in the field of centrifugal force,

4) The Svedverg and J. B. Nichols, *J. Am. Chem. Soc.*, 45, 2910 (1923)
The Svedverg and H. Rinde, *ibid.*, 46, 2677 (1924)

5) S. Odén, *Proc. Roy. Soc. Edinburgh*, 36, 219 (1916)
The Svedverg and H. Rinde, *J. Am. Chem. Soc.*, 45, 943 (1923)
E. O. Kraemer and A. J. Stamm, *ibid.*, 46, 2709 (1924)

$$r=\sqrt{\frac{9\eta\ln(x_t/x_0)}{2(d_p-d_m)\omega^2 t}},$$

where d_p and d_m are the density of disperse phase and medium respectively, η is the viscosity of medium, ω the angular velocity, x_0 the distance between the rotation axis and the bottom of rotating vessel, x_t the distance between the rotation axis and the boundary after time t.

In order to determine the distribution of particle size, the thickness of the oil layer separated, at first, is measured with time. Next, the thickness of the layer is converted into the percentage referring to the total quantity of disperse phase, and the percentage is plotted against time (accumulation curve). And then the tangents of the curve referring to time are drawn, and the value of Δp between the cross point of each tangent and the percentage-axis is obtained. On the other hand the radius corresponding is calculated from the above equation and the distribution diagram (Fig. 2) is obtained. The area of each rectangle, $\left(\frac{\Delta p}{\Delta r}\times\Delta r\right)$, in the

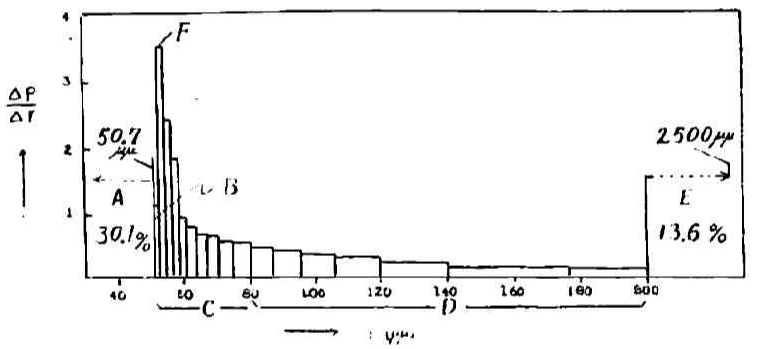

Fig. 2 (Exp. No. 7) Distribution diagram.

diagram, accordingly, shows the percentage of the quantity of disperse phase existing between each radius for the total quantity of disperse phase.

In order to convert the thickness of the oil layer at each time into the percentage for the total quantity of disperse phase, the quantity of oil in the portion below the oil layer and in the total portion are measured by evaporating water and weighing the residual. In this case the quantity of oil is calculated considering that the emulsifying agent, lecithin, is equally soluble in both oil and water*.

* From the result that the stability of emulsion is exceedingly less when the emulsifying agent is less than about 1%, it is certain that the agent is more soluble in oil than in water. Accordingly, in this case, the quantity of oil in the portion below the oil layer is much more than the result above mentioned. If the calculation is done in the case of Exp. No. 7 regarding all the agent dissolved in oil, the quantity of small particles of oil remained below without coagulation becomes to 37.7% which is a larger value in comparison with 30.1% obtained in the above consideration of equi-distribution.

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Experimental results.

The distribution diagrams under various conditions are obtained by the procedure above described, and Fig. 2 is one of these examples. The effects under various conditions will be shown in Table 2, being obtained from the distribution diagrams.

Table 2

Exp. No.	A (%)	B (μμ)	C (%)	D (%)	E (%)	F (μμ)	F (%)
1	22.7	137.5	...	23.0	54.3	154.5~180.1	12.5
2	23.4	78.4	5.3	56.7	14.6	78.4~ 81.5	24.2
3	22.8	58.6	24.1	33.3	19.8	58.6~ 61.1	12.0
4	29.2	49.9	34.4	24.3	12.1	49.9~ 51.3	8.7
5	20.4	63.8	27.6	37.0	15.0	63.8~ 67.0	13.4
6	27.9	53.7	30.0	29.6	12.5	53.7~ 55.5	7.2
7	30.1	50.7	30.0	26.3	13.6	52.3~ 53.9	5.6

A: % of particles without coagulation
B: Minimum radius of coagulation particles
C: % of particles between minimum radius of coagulation particles and 80 μμ
D: % of particles between 80 (137.5) and 200 μμ
Where the value, 137.5 μμ, is only in Exp. No. 1.
E: % of particles between 200 μμ and maximum radius
Where the value of maximum radius is 50 μ in Exp. No. 1 and is 2.5 μ in others.
F: Location of the maximum distribution of particle size and its %

The authors express hearty thanks to the Ministry of Education for the Scientific Reserch Grant.

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